## National Burgh of Standards Certificate of Analysis

## Standard Reference Material 951 Boric Acid

| H <sub>3</sub> BO <sub>3</sub> , acidimetric assay, weight percent | 100.00 | $\pm 0.01$ |
|--|--------|------------|
| Absolute Abundance Ratio, <sup>10</sup> B/ <sup>11</sup> B         | 0.2473 | ± 0.0002   |
| Boron-10, atom percent   | 19.827 | ± 0.013    |
| Boron-11, atom percent   | 80,173 | ± 0.013    |

The atomic weight of the boron, calculated from the absolute abundance ratio using the nuclidic masses 10.0129 and 11.0093, is 10.812.

This lot of boric acid was prepared to ensure material of high purity and homogeneity. As received, it was slightly deficient (approximately 0.01 percent) in moisture, but adjusts to a stoichiometric composition in about 30 minutes exposure to a normal room humidity (approximately 35 percent relative humidity). Once adjusted to composition, the material is relatively insensitive (<0.01 percent) to moisture changes between 0 and 60 percent relative humidity, and absorbs only about 0.02 percent excess moisture in room temperature humidities as high as 90 percent. The material cannot be heated as it decomposes with the loss of considerable water.

Assay was by coulometric titration of samples varying in size from 0.2 to 1.0 g of boric acid, dissolved in 100 ml of a preneutralized solution 1M in KCl and 0.75M in mannitol. The inflection point of the potentiometric curve obtained from measurements with a glass-calomel electrode system was taken as the end point. The pH of the maximum inflection point was taken as the end point. The pH of the maximum inflection point will vary from approximately 7.9 to 8.5 for the range of sample sizes given above, and the titration must, therefore, be conducted in the absence of carbon dioxide or carbonates. The indicated tolerance is at least as large as the 95 percent confidence level for a single determination of any sample in the lot of material, and the average essentially indicates a boron-hydrogen ion ratio of 1.0000, since separate examination shows the material contains less than 0.001 percent of free strong acid.

The abundance ratio was determined by single-filament solid-sample mass spectrometry using the ion Na<sub>2</sub>BO<sub>2</sub>. Mixtures of known <sup>10</sup>B/<sup>11</sup>B ratio (at a 1:4, 1:1, and 4:1 ratio) were prepared from high-purity separated isotope solutions and used as comparison standards. Correction was determined for the <sup>16</sup>O/<sup>17</sup>O ratio (<sup>11</sup>B/<sup>10</sup>B ratio correction, -0.00079) by measuring mass 91 using the high-purity boron-11 separated isotope. The indicated tolerances are at least as large as the 95 percent confidence limits for a single determination which includes terms for inhomogeneities in the material as well as analytical error.

The material was prepared by the J. T. Baker Company of Phillipsburg, New Jersey, for the Argonne National Laboratory. Separated isotopes were purified and solutions prepared by K. M. Sappenfield and T. J. Murphy, coulometric titrations were made by G. Marinenko and C. E. Champion, mass-spectrometric measurements were made by E. J. Catanzaro and E. L. Garner, Analytical Chemistry Division. The various procedures developed have been published and are available in Special Publication 260-17.

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of W. R. Shields.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by J. L. Hague.

Washington, D. C. 20234 February 28, 1969

W. Wayne Meinke, Chief Office of Standard Reference Materials